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RESEARCH MEMORANDUM

THERMAL STABILITY OF A COMMERCIAL PROPYL PENTABORANE (HEF-2) IN THE RANGE 147° TO 190° C

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Cleveland, Ohio

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RESEARCH MEMORANDUM

THERMAL STABILITY OF A COMMERCIAL PROPYL PENTABORANE (HEF-2) IN THE RANGE 147° TO 190° C

By G. McDonald

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SUMMARY

The thermal stability of a commercial grade of propyl pentaborane (HEF-2) was investigated at temperatures of 147° and 190° C. The products of decomposition are hydrogen, hydrocarbons, and a solid nonvolatile yellow boron hydride.

The following equation was developed from which may be calculated the approximate time required to decompose 20 percent of propyl pentaborane at any other temperature:

$$\log(t_{20\%} - 2) = \frac{7.85 \times 10^3}{T} - 16.3$$

where T is temperature in °K and $t_{20\%}$ is the time in minutes required for decomposition of 20 percent of the sample.

INTRODUCTION

A study of the thermal stabilities of decaborane and of ethyl decaborane (HEF-3) was previously completed at the NACA Lewis laboratory (ref. 1) in order to furnish data pertinent to fuel-system design and fuel storage. The study reported herein was made to obtain knowledge of the thermal stability of commercial propyl pentaborane (HEF-2) and to compare this stability with the previously measured stability of pentaborane (ref. 2) and decaborane (ref. 1). Measurements were made of the amount of nonvolatile decomposition products formed when propyl pentaborane was heated for various times at temperatures of 147° and 190° C.

APPARATUS, MATERIALS, AND PROCEDURE

The propyl pentaborane was heated in a cylindrical stainless-steel bomb 3 inches long having an outside diameter of 5/8 inch and an inside

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diameter of 11/32 inch. The volume was approximately 1/4 cubic inch. The bomb was closed with a 1/8-inch stainless-steel Hoke valve and heated in an aluminum block furnace, which fitted closely about the bomb. The furnace was electrically heated and suitably insulated. A thermocouple well extended through the block to the midpoint on the bomb.

The propyl pentaborane used was of commercial grade and consisted of approximately 80 percent monopropyl pentaborane, 15 percent dipropyl pentaborane, and 5 percent pentaborane. The material was used without further purification.

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The HEF-2 or propyl pentaborane (1.4 g) was placed directly in the weighed bomb. This transfer was performed in an inert atmosphere. The bomb was sealed, evacuated, weighed, and placed in the furnace for the desired time. After removal from the furnace, the bomb was quenched and the volatile material was removed at a temperature of 110° C on a vacuum system until constant weight was obtained. The distillation temperature was below the temperature of decomposition. The bomb was reweighed to determine the nonvolatile residue. The percent formation of nonvolatile residue, for use in figures 1 and 2, was calculated by dividing the weight of nonvolatile residue by the weight of initial fuel sample. The percent of decomposition used for the calculations and for figure 3 was determined from the ratio of the percent of nonvolatile residue at any time to the percent of nonvolatile residue at a time sufficient for complete decomposition.

RESULTS AND DISCUSSION

A curve of the percent of formation of nonvolatile residue plotted against time is shown in figure 1 for propyl pentaborane at a temperature of 147° C. Similarly to pentaborane (ref. 2), the propyl pentaborane shows a decrease in rate of decomposition with time. This relation is in contrast to the nearly steady rate of formation of nonvolatile residue, with increase in time, which was observed with decaborane and commercial ethyl decaborane (HEF-3) (ref. 1). A curve for the percent formation of nonvolatile residue plotted against time at a temperature of 190° C for propyl pentaborane is shown in figure 2. This curve at 190° C shows a shape similar to that of the curve that was obtained at 147° C. Experiments at both temperatures were carried out for a time sufficient to decompose the sample completely.

Figure 3 shows a plot of the reciprocal of Kelvin temperature against the $\log(t_{20\%} - 2)$ where $t_{20\%}$ is the time in minutes required for decomposition of 20 percent of the sample. Two minutes has been subtracted from the time required for the decomposition of 20 percent of the sample in order to remove from the calculations the approximate time required to bring the sample up to reaction temperature (ref. 2).

These data for propyl pentaborane may be represented by the following equation:

$$\log(t_{20\%} - 2) = \frac{7.85 \times 10^3}{T} - 16.3$$

where T is temperature in $^{\circ}K$. The percent decomposition of propyl pentaborane is not a linear function of time, in contrast to the decomposition observed for decaborane. However, for percentages of nonvolatile residue less than about 20 percent, the time required to form any given percent of nonvolatile residue may be approximated by assuming a linear relation between percent formation of nonvolatile residue and time.

The physical properties of the residue that is obtained from propyl pentaborane are similar to those obtained from the pyrolysis of pentaborane both in color and lack of a softening or melting point. While the residue from propyl pentaborane was not investigated as completely as was the residue from decaborane and ethyl decaborane (HEF-3), it is expected from the characteristics observed that the residue is not extensively soluble in boron hydrides or alkylated boron hydrides. The extent of fuel-line clogging with propyl pentaborane might be expected to be somewhat more than that observed with pentaborane because of the decreased stability. As observed from figure 3, the stability of propyl pentaborane is less than that of pentaborane, decaborane, or ethyl decaborane. Similar to the stability relation between ethyl decaborane and decaborane, propyl pentaborane is less stable than pentaborane.

Similar to the experience with pentaborane, pressure buildup occurs with propyl pentaborane because of the liberation of a mixture of hydrogen and hydrocarbons. Below 100° C, pressure buildup is slow and at normal temperature of storage is sufficiently slow to permit storage to be a practical operation.

CONCLUSIONS

The results indicate that a commercial grade of propyl pentaborane (HEF-2) is less stable to pyrolysis than pentaborane. Physical properties of the product of pyrolysis are similar to those of the product of pyrolysis of pentaborane.

Lewis Flight Propulsion Laboratory
National Advisory Committee for Aeronautics
Cleveland, Ohio, August 30, 1957

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2. McDonald, G. E.: Thermal Stability of Pentaborane in the Range 329° to 419° F. NACA RM E54G16, 1956.

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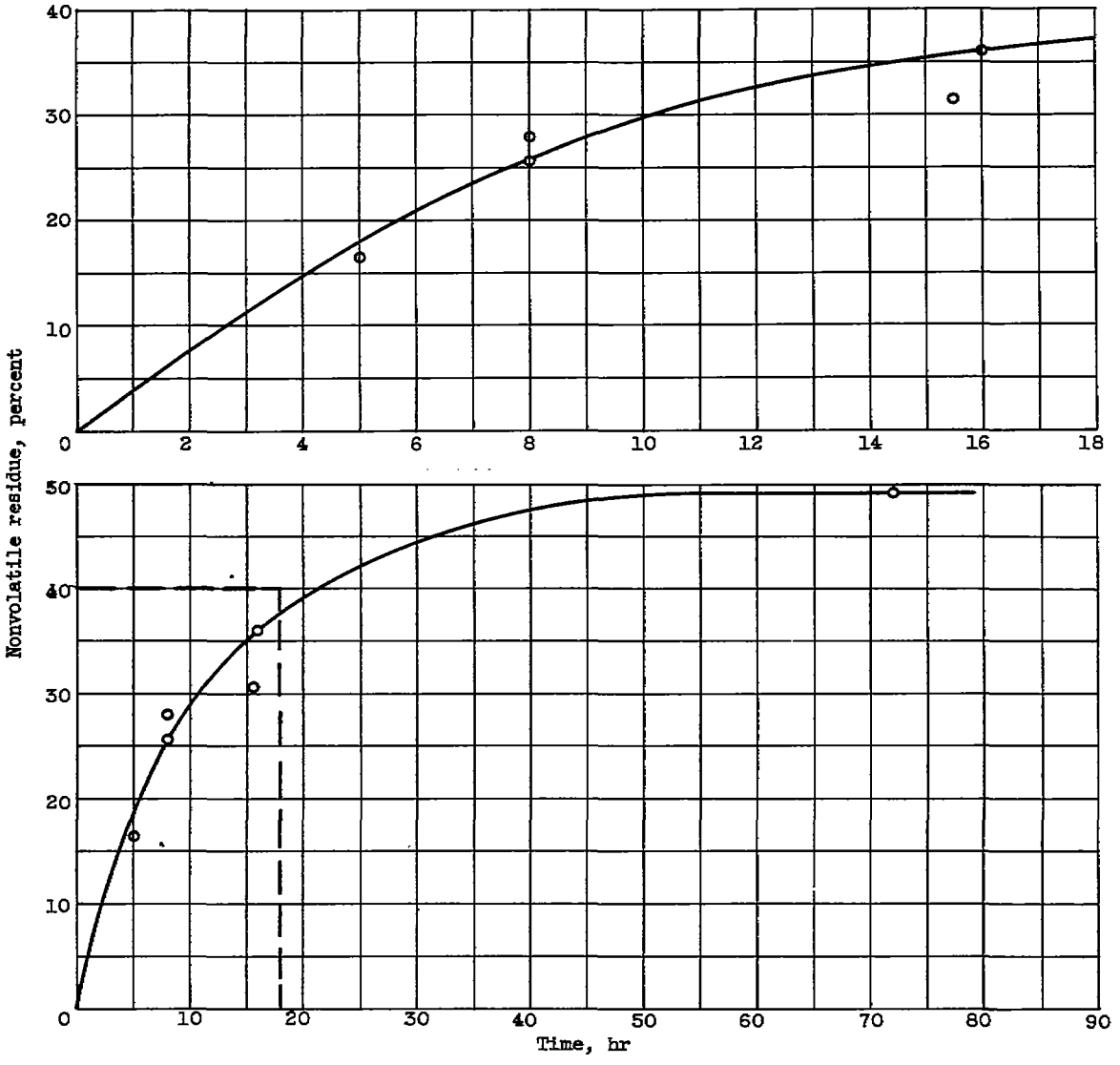

Figure 1. - Decomposition of propyl pentaborane at 147° C.

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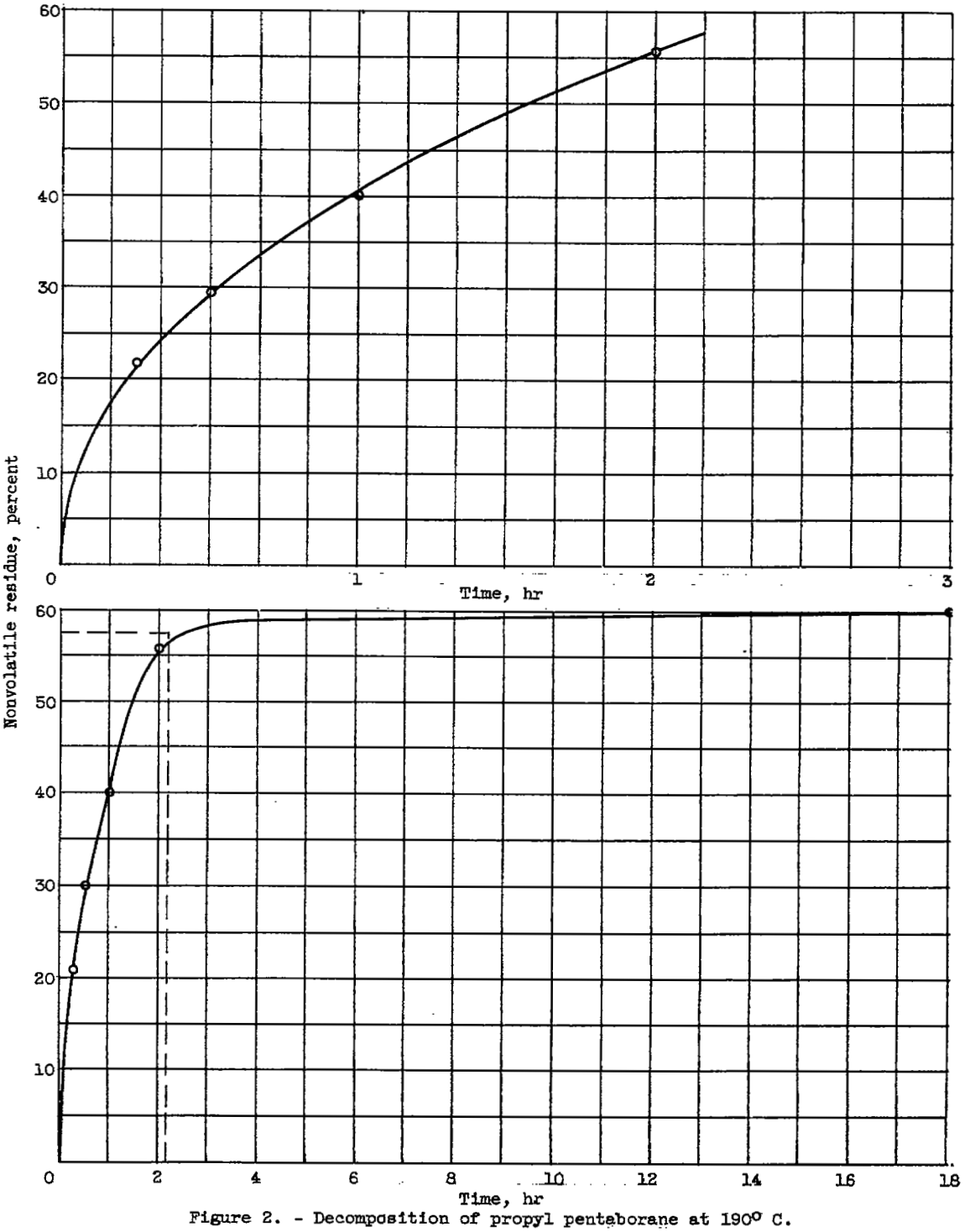

Figure 2. - Decomposition of propyl pentaborane at 190° C.

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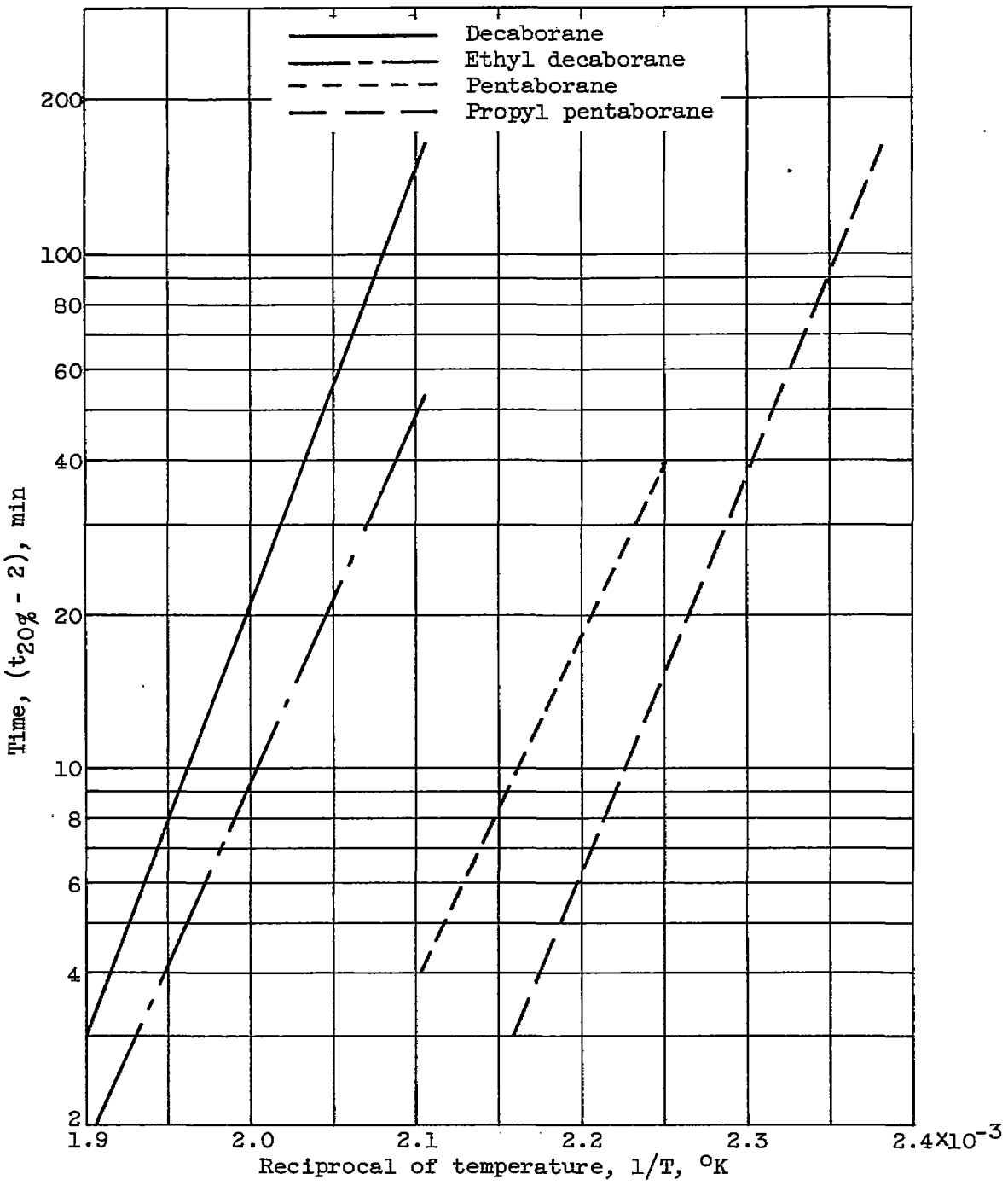

Figure 3. - Reciprocal temperature plotted against time required for 20 percent decomposition.

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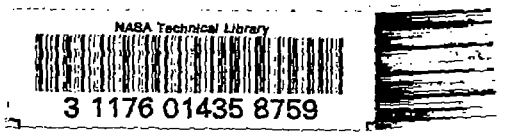

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